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# MECHANICAL AND LINEAR VISCOELASTIC PROPERTIES OF HIGH DENSITY POLYETHYLENE OBTAINED FROM TENSILE AND DEAD-LOAD CREEP TESTS

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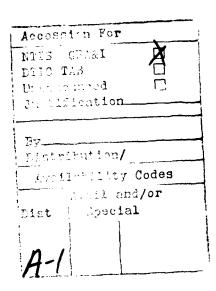
#### **ABSTRACT**

Three grades of high density polyethylene (HDPE) are tested for tensile, creep and creep recovery, and creep properties determination. As a result of tensile tests, the materials were found to strain uniformly under constant strain rate to about 3% at 20°C which is well below the 12% yield point (YP). With creep tests, an attempt was made to explain and predict the effects of low loads and temperature on linear viscoelasticity (LVE) in these materials. When a creep curve plateaus (zero creep strain after a transient primary creep strain), strain recovery from the plateau is 100%. This combination of creep plateau and full recovery as another manifestation and definition of linear viscoelasticity is presented. The effort presented was only partially successful for HDPE as the creep apparatus has an error zone producing uncertain data precisely in the region where HDPE linear viscoelasticity is expected. However, from past research on the same creep apparatus, it was found that amorphous polycarbonate (PC) produces plateaus outside and above this error zone, from which full recovery occurs at strains up to 1% and stresses up to approximately 3300 psi (22.75 MPa). An engineering load-strain data base is established for the three grades of HDPE tested using the U.S. Army Materials and Mechanics Research Center (AMMRC) streamline tensile specimens. A comparison is made to the load-strain results from two types of ASTM tensile specimens. It is noted the AMMRC streamline tensile specimens have a separate response to mechanical tensile loading from that of ASTM-type specimens so that care must be exercised when comparing their mechanical performance to that of ASTM-type specimens.

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## INTRODUCTION

Until there is confidence in performing materials characterization to tailor the material to the job, the only choice is to defer to the state-of-the-art approach exemplified by industry as, for example, by the General Electric Technology Center, the Davidson Rubber Company, the Monsanto Chemical Company's Applications Development Center, the Borg-Warner Design Engineering Laboratory, and the Dow Chemical Company's Materials Engineering Center. For example, General Electric has customer-accessible materials computer programs from accumulated data bases containing information on the mechanical and physical properties so that through and with such data more economical and efficient use of finite element computer programs can be made to aid in solving the end product problems of the materials-utilizing customers.

The need to generate precise and reproducible data bases is recognized, and the work done by producers of raw plastics in their attempts at producing data bases from various mechanical tests should be applauded. These data bases aid in solving actual structual problems and locating defects in end products manufactured by users of their raw plastics.<sup>2-4</sup>

There is also a need to extend the existing quality data bases of the marketable shelf materials of the producers/suppliers into the tensile test region of small loads and strains along with corresponding creep tests. However, increasing the precision of the tensile test and, thus, the data accuracy in the low-load region alone would not solve the problem of a plastic structure under creep as, still, only short-time loading data is produced. However, low-load creep data, at or below the critical stress level where creep is minimal or nil, would extend the present state-of-the-art tensile test data bases of the producers/suppliers and help them in reaching the ultimate goals (needs) of the end products fabricators: tailor made materials, efficiently obtained, through materials characterization.

The purpose of the present work is to establish a data base for the three grades of HDPE. (tensile and creep) over a range of loads and temperatures. The tensile tests are restricted to 12% strain, and the creep tests to low dead loads and all testing is further restricted to the AMMRC streamline (SL) tensile specimens.<sup>5</sup> These tests results are compared with those from two ASTM-type specimens. It is shown that the ASTM and SL specimens produce different mechanical properties so that caution must be exercised when selecting specimens.

It is further shown that the tensile test not only supplies engineering data, but can be a useful tool in solving problems related to more fundamental properties of plastics. It is also shown that there is a load-strain cutoff point below which strain is uniform and strain rate is linear (constant). This cutoff is to related to work performed by Moore and Matsuoka.<sup>6</sup>

The creep tests resulted in very flat curves suggesting a plateau for strain with time such that the creep rate is zero. The creep rate reaches a maximum during dead loading (at full load) and decreases in the transient, primary creep region, to gradually become zero on the strain plateau. Nielsen<sup>7,8</sup> indicated a critical stress for HDPE below which creep practically ceases (about 3.5 MPa or 500 psi). Hence, an objective of this study is to determine whether creep testing at low loads below the critical stress indicates a linear viscoelastic (LVE) region and whether the plateau concept has any relationship to Nielsen's critical stress.

It is believed that more attention is needed in a sorely neglected region of mechanical testing, (called by us as the **loading problem**): the initial time-load-strain region during mechanical loading of a tensile specimen and for a short time after full load (a region

common to all mechanical tests). Further, the initial loading is not instantaneous but takes a finite, measurable time. Since plastics are sensitive to temperature and strain rate, the behavior of their initial loading region in the presence of these parameters affects their long-time response. It is worthwhile to note that the low-load creep test and the low-load tensile test could follow equivalent loading paths for the specimen. It is also worthwhile noting that the loading problem exists for metals 10,11 so that it can be considered, more generally, a materials problem.

# MATERIALS, SPECIMENS, AND EXPERIMENTAL PROCEDURES

The material tested consists of three grades of high density polyethlene produced by Phillips 66 Company in granular form. The designations for the materials are: EHM 6001 as sheet grade (SG), HHM TR 140 as blown-film grade (BFG), and HXM 50100 as the container material. There are three sources of HXM 50100 samples as tested. The earlier natural-colored container (NCC) and the later green-colored container (GCC) with 4% by weight green pigment<sup>12</sup> are two of these sources. Both of these contained about 20% regrind to the best of our knowledge. The containers, NCC and GCC, were made by a subsidiary of the Ford Motor Company using an unidentified blown-film process. They added the 20% regrind to HXM 50100 (presumably the same grade). The third source was virgin granular material from which three compression-molded plates (1/4 x 12 x 12)in.3 were made on a 100-ton press13 with integral heating and cooling controls according to ASTM standard but at three different cooling rates: 3.3°F/min, air cooled (Plate I); 30°F/min, air and tap-water cooled (Plate III); and at 90°F/min, tap-water cooled (Plate II). The slowest cooled is expected to have the highest crystallinity content. Plate III was cooled near the ASTM specified rate. Upon cooling, all three plates showed shrinkage. as well as slight thickness variation determined on tensile specimens cut from the plates during micrometer measurements of the gage length (GL) cross section.

The molecular weights from the Phillips 66 Company's materials literature, along with melt-flow index values, are tabulated in Table 1 along with our values obtained from the gel permeation chromatography (GPC). The GPC values are from untested injection-molded specimens which contain no regrind and the HXM 50100 is from the NCC and not from virgin pellets.

Table 1. CHEMICAL ANALYSIS OF THREE GRADES OF HIGH DENSITY POLYETHYLENE

		Molecula	ar Weight			
Designation	Specimen	Mw Weight Average	Mn Number Average	Mw/Mn	Melt Flow Index*↑	HLMI (F)*‡
EHM-6001	Phillips*	156,000	19,100	8.16	0.15	24**
	Ours	120,000	17,700	6.80	-	_
HHM-TR140	Phillips	177,000	16,400	10.80	0.28	-
	Ours	130,000	11,200	11.60	-	_
HXM-50100	Phillips	183,000	19,900	9.20	0.07**	10
	Ours	153,000	18,460	8.15	-	_

<sup>\*</sup>Phillips 66 Company

<sup>†</sup>g/10 min ‡10g/10 min

<sup>\*\*</sup>Estimated Values

The SG and BFG specimens were injection molded at 246°C. Thermogravimetric tests (not shown) on the materials at a heating rate of 10°C per minute in air indicate the start of degradation at 246°C [an unzipping of the polyethylene (PE) molecules] which could affect their molecular weights and, hence, their mechanical properties if processing time is extended, which was not the case here. <sup>16</sup>

An injection molding machine<sup>17</sup> was used on the SG and BFG grades (see Table 2) with a mold that made specimens with five shapes simultaneously per mold cycle. Three of these shapes were used for testing. Two of the shapes are bars 5 inches long by 1/2 inch wide, with thicknesses of 1/8 inch and 1/4 inch. The third is that of a dogbone similar to ASTM Type I<sup>18</sup> with 8-inch overall length, 1/8 inch thick with 1/2 inch uniform width along a 2.5-inch gage length. These bars and dogbones have a slight end-to-end taper in thickness. In addition, there is material shrinkage and uneven cooling causing bar specimen faces to be somewhat concave, more so in the 1/4-inch bars, a slight curvature both perpendicular and parallel to the faces of the bars, and probably some material orientation of unknown detail and amount.

From the 1/8-inch-thick bars, tensile specimens were routed similar to ASTM Type  $IV^{18}$  to a width tolerance of  $\pm 0.005$  inch along a 1-inch GL. The width tolerance, longitudinal thickness taper, concavity of sample faces, and extensometer **knife**-edges results in cross-sectional variations in the 1-inch GL. The combination of these effects caused undesirable necking at the ends of the 1-inch GL with sufficient frequency to cause abandonment of extensive testing with the ASTM Types I and IV dogbone specimens except for some testing for comparison and control purposes.

As a result, the AMMRC SL tensile specimen was chosen for testing.<sup>5</sup> The SL shape was found to be useful for tensile testing soft plastics like HDPE, or any plastic softened by sufficiently high test temperatures where the extensometer knife-edge pressure and/or existing variations in specimen cross section causes stress concentrators in the cross section. The streamline taper tends to minimize these effects except near melting in HDPE; i.e., greater than 120°C.

A pantograph was used to make SL tensile specimens from both bar thickness of the SG and BFG grades, from 1/4-inch-thick pieces cut from the NCC and GCC containers, and from the three compression-molded plates of HXM 50100.

Specimens with the SL design were exclusively used for dead-load creep and tensile testing. Exceptions are SG (1/2 in. x 1/4 in.) x (5-1/2 in.) bars used supplementarily with low loads roughly of 350 psi or less. A silhouette of the SL specimens with dimensions is shown in Figure 1. The 1-inch GL of the SL specimen has a 1/2-inch straight-sided middle section, flanked by tapered 1/4-inch parts.

In all the mechanical testing, the SL specimens were gripped such that the initial length of specimen between grips [Head Distance (HD) (time = 0)] is 3 inches with a 1-inch extensometer centrally located. The 1/2-inch-long straight-sided, central part of the GL is 1/4 inch wide. The 1/4-inch-long tapered parts, one on each side of the 1/2-inch straight-sided central part, are also very close in width, along their lengths, to the 1/4-inch-wide central part, due to the ±0.005-inch machining tolerances overlapping the taper dimensions in the GL. This makes the GL nearly straight-sided in these small SL specimens. Yet, at the ends of the 1-inch GL, and just outside it, the taper exceeds the machining tolerance sufficiently so as to have a larger cross section there to counteract the extensometer knife-edge indentations and overcome the effects of stress concentration. Then the strain near and at necking tends to be confined inside the 1-inch gage length.

Table 2. PRODUCTION SETTINGS ON VAN DORN 200 INJECTION MOLDING MACHINE USED FOR PRODUCTION OF HIGH DENSITY POLYETHYLENE (MARLEX)\* SPECIMENS

					Tempera	Femperature (°C)				Presssure (psi)	e (psi)			Timer (sec)	(sec)			Ma	Machine Settings	<b>s</b> 6t	
HDPE Grade	Melt Index Density (g/10 min) (g/cc)	Density (g/cc)	Nozzle	Front	Front Middle Rear Cavity Amblent	Rear	Cavity	Amblent	Boost	Boost Injection Hold Back Injection Hold Delay Cycle	Hold	Back	Injection	Hold	Delay		Shot Size	Weight (9)	Weight No. Injection (g) (pts./shot) Speed		Screw Speed
EHM-6001	0.15	0.960	232.2	246.1	232.2	215.6	215.6 29.2 19.4	19.4	675	750	450	8	4	11	23	38 1.60 77.4	1.60	77.4	r)	Fig	110
HHM-TR140 0.28	0.28	0.947	232.2 246.1	246.1	232.2	215.6	28.3	21.1	0//	850	450	50	4	=	22	38 1.65	į	0.77	S	Full	110
*Phillips 66 Company	Sompany																				

Small, superficial nicks and/or scratches on the specimen edges or faces did not affect the test results, nor did sanding some of the edges of the routed SL specimen contours. Throughout this report, in figures and text, when referring to the test on a particular type specimen and its HDPE grade, we use the notation:

(GRADE)(GL-AREA)(SPEC. TYPE)(TEST TYPE)(HS, in./min)(TEST TEMPERATURE).

On occasion, parts of this notation are left out when the meaning is obvious or redundant. For example:  $(SG)(1/8 \times 1/4)(SL)(TT)(5.0 \text{ in./min})(70^{\circ}\text{C})$  might be reduced to  $(SG)(1/8 \times 1/4)(SL)$  if, from the text, the test is obvious. The **GL-AREA** is the cross-sectional area of the gage length, and is given as a product of thickness multiplied by width in square inches (implied).

The apparatus used for creep testing was a four-station creep unit 19 with an environmental chamber (EC)<sup>20</sup> as an integral part. Only one station was used. Each station has a maximum dead load (DL) of 500 lbf. The desired DL is made up from an assortment of weights from 1/10 lbf to 40 lbf. The sample, extensometer, weights, and all rods and couplings of the load train are in line and suspended from a U-frame welded at its ends to the steel frame of the creep apparatus. Upon starting, after a sudden initial impulse, recorded on the e versus t strip-chart curve because the load platform is attached to the structure supporting the specimen, (see the discussion of the experimental strain error in this section), the load is applied smoothly through an electric motor coupled to a worm gear moving a cantilevered elevator platform (upon which the load rests) at approximately 5 inches per minute up or down. The loading of a specimen involves a downward motion of the elevator until slack in the coupling is taken up and the DL is subsequently airborne. The elevator stops automatically after an arbitrarily preset, noncritical space (adjustable from 0 to 1 inch by means of a proximity switch between the bottom of the flat weight pan and the floor of the elevator platform) is achieved. The upper part of the stainless steel extensometer, with the flat sample, is located in the EC while the lower part, with a linear variable differential transformer (LVDT)<sup>21</sup> for precision measurement of strain and adjusting micrometer, is outside and below the EC and just above the DL. Figures 2 and 3 (pages 303 and 313) are the photos in the Metals Handbook<sup>22</sup> of the typical rod and tube-type extensometer that were used with adaptation for flat specimens.

The chapters on creep stress rupture and stress relaxation testing in the Metals Handbook<sup>22</sup> describe the procedures used in preparing and loading for the creep test, a measure of strain in real time (at constant DL and constant test temperature). The extensorieter measures the sample's thermal contraction/expansion while the sample is in an unloaded state. Only the intrinsic weight of parts of the load train act upon the sample when cooling/heating from ambient to a lower/higher temperature. The LVDT is rated for 0.25-inch full run-out which corresponds to a 10-volt strain extension (it is possible to find linearity on a side of null in the LVDT to 16 volts or 40% strain in a 1-inch GL).

A strip-chart recorder<sup>23</sup> with a chart speed of 1 to 20 in./min and 1 to 20 in./hour and a voltage strain-scale range of 1 millivolt to 10 volts is used to record the strain output of the creep test. By halving the voltage scale range by a multiplier, 20 volts per 10 inches of strain scale is obtained on which the maximum strain of 16 volts (40%) can be read and recorded using a 1-inch GL.

A microprocessor for controlling temperature and temperature rates was hardwired to the EC obtained through Associated Environmental Systems. It controls the liquid nitrogen solenoid switch and the 1200-watt heater by monitoring a thermistor near the sources of heat and cold which are hidden behind a stainless steel baffle and are distributed to and mixed within the EC by a constant speed fan. The stainless steel low-temperature EC can be cooled close to liquid nitrogen temperatures with special rubber seals on the door which is the front side of the EC. It can also be heated to approximately 190°C.

Three temperature sensors were used in the EC. The **buried** one, in the back of the chamber (in the fan housing close to the heater and close to cold nitrogen gas/liquid jet from the outlet of the solenoid controlling the liquid nitrogen) was the thermistor element hardwired to the chamber. It produced visual read-outs on the chamber control panel and microprocessor control panel. The other two sensors are Type K thermocouples (TC), one free to be positioned in a cavity of the chamber between the fan and sample and the other touching on a flat surface of the specimen gage length facing the fan. The digital temperature read-outs from the Type K TCs were found to lag by 1.5°C. The microprocessor digital temperature read-out from the thermistor on both specimen cooling and heating, and the TC on the specimen face had a minus 1/2°C shift in a digital read-out zero point. These were accounted for in the final results. The 1.5°C lag was due to the temperature gradient between the two sensor positions in the chamber: the **buried** thermistor, near the source of heating and cooling, and the specimen TC both dependent on the fan for convected temperature.

A digital voltmeter with read-out to four decimal places was used to calibrate the LVDT zero point and the desired voltage range on the strip-chart recorder.

The lower part of the extensometer (and lower specimen grip and grip rod) acted as an **intrinsic** weight of approximately 3 lbf DL. At room temperature  $(21^{\circ}\text{C} \pm 1^{\circ}\text{C})$ , the starting point for all experiments, the zero-point strain was adjusted with the intrinsic load present on the specimen. Thus, as the sample contracted or expanded with temperature, these thermal changes were modified by the tensile strain due to the intrinsic 3-lbf load (see Figures 4 and 5).

The creep apparatus, as described, was not able to provide error-free data at or below 2.41 MPa (350 psi) because the extensometer exerted a small bending moment on the flat specimen upon loading and unloading which introduced strain errors in the creep and recovery strains sufficient to negate test results.

The bending moment is due to the mounted specimen and extensometer. Together they deviate from vertical in the load train of the creep apparatus prior to application of the external load. When the external load was applied, the bending moment, while bringing the specimen and extensometer in line with the load train, produced error in strain.

The errors were particularly noticeable in strains from small loads where full-scale strain on the chart recorder was set to read a maximum strain of 0.0025 in./in. or less. Upon applying or removing a DL (however smoothly) by way of the receding elevator, capricious strains were introduced due to lateral movement of parts of the load train. This produced bending moments on the specimen. They were capricious in that the bending moments appear to occur in different combinations (for each new specimen tested) upon each loading/unloading. Further, due to processing, all specimens had a slight curvature along the length which was another source of random, capricious strain.

There was also an independent jump in positive or negative strain at the instant the elevator was activated, due to the elevator drive motor being connected to the creep frame. An undesireable, impulsive force was transmitted to the load train causing a negative shift in zero-strain setting at the start of DL application and a positive shift just prior to unloading. These two impulsive forces caused a permanent shift in zero-strain position which is opposite to the direction of application of the DL. These impulsively induced strains were sufficiently large to be important for creep tests with small DLs and their consequent small strains. Furthermore, they were also found to be capricious as their amplitudes varied in a random way with any load or unload application.

All the creep recovery curves involved loads with external weights, and had a strain uncertainty of about ±0.0005 in./in. at 20°C and about double this at 70°C. The uncertainty occurred at both loading and unloading and may have reinforced or cancelled each other capriciously so that a maximum uncertainty of  $\pm 0.002$  in./in. is possible at  $70^{\circ}$ C. This accounts for some of the confusion that the creep recovery runs presented before the role of this capricious strain error was more fully recognized. As the strain in the recovery tail (at 70°C) of the creep recovery curve of Figure 6 was near 0.00225 in./in., the error in recovery strain could be near 100%. We emphasize that the creep apparatus, described herein, was perfectly acceptable once these errors became negligible for the load and strain scale of the experiment. The author, A. A. Warnas, performed a series of creep tests on both HDPE and polycarbonate<sup>24</sup> over a wide range of loads and temperatures that were outside the range of these errors with no ill effects from them. None of these arguments affected the measurement of thermal strain (due to the coefficient of linear expansion) when only accompanied by the thermal strain from the ever-present intrinsic loads acting upon a specimen. There was neither application of external loads in the form of dead weights, nor was there any motion of the elevator to induce an impulsive force to the load train to perturb the strain. The remaining testing procedure consisted of zeroing the extensometer at room temperature and producing a temperature ramp, up or down, to the desired end temperature (see the Intrinsic Creep Loads and Specimen Heating at Constant Temperature Rate Section).

# **Tensile Equipment**

The equipment used for the tensile test (TT) was a 20 kip Instron universal electrome-chanical test machine of constant crosshead speed with Instron clip-on-type extensometer<sup>25</sup> and an environmental chamber (EC). The Instron had 20 kip, hand-tightened grips with bottom grip spring loaded.

A general description of this type of machine and procedures for testing with it is given in Metals Handbook,<sup>22</sup> pages 28 to 31, 34 to 37, and 47 to 51 with appropriate adaptations for plastic kept in mind.

In the present setup, the EC works automatically in controlling 'emperatures at  $21^{\circ}$ C and above, however, due to a faulty relay, it has to be controlled from the temperature TC readout at temperatures below  $21^{\circ}$ C (with a  $\pm 4^{\circ}$ C deviation in temperature) by manually controlling the liquid and gas nitrogen input. Both methods of temperature control relied on the

signals from a TC touching on a GL face of the specimen. One to two TT runs were performed at temperatures below 20°C for each test temperature.

At  $20^{\circ}$ C  $\pm$   $2^{\circ}$ C, without the EC, two to four tests were performed for each head speed from 0.005 to 5 in./min. By means of two identical X-Y recorders, all TTs simultaneously produced load versus strain and load versus time. These were later converted to strain versus time data by eliminating the load between them. For tests in Figures 1, 7, and 8, a 1 inch, 100% strain extensometer was used. For all others, a 1 inch, 10% extensometer was used (linear to 12%). Data with TT strains greater than 12% refer to the 100% extensometer.

Prior to a TT, the most sensitive load and strain scales were used to eliminate inadvertent preloads on a mounted specimen due to specimen-gripping strains and specimen contraction at low temperature or expansion at high testing temperature. The manual control crosshead-motion knob on the tensile machine was used to damp out the preload and zero the load cell. At this time, the extensometer was rezeroed.

# **Experimental Results**

TT and Tensile Creep

Figures 1a and 1b show the TT engineering load-strain curves for SG and BFG HDPE at 21°C and HSs from 0.005 to 5 inches per minute. They indicate a clear superiority of SG over BFG in terms of Young's modulus and higher yield point (YP) at all HSs.

The YP stress of both grades is sensitive to HS with the YP strain decreasing as HS increases. The arrows on the curves indicate the location of the YPs (also shown in Figure 9).

The dash lines in Figure 1a are engineering load-strain curves for (SG)(1/4 x 1/4) (SL)(21°C). These specimens, with double the GL cross section but the same silhouette geometry (superimposed for comparison on the data of the (SG)(1/8 x 1/4) (SL)(21°C) specimens) indicate no load doubling at a given strain at their respective HSs. [In TTs, with HS and temperature constant (performed with Types I and IV ASTM specimens at any given strain up to yield) if the cross-sectional area of the GL is doubled, then the load is doubled (see Figures 9a and 9b)].

Since there is not a relationship between SL specimens which are identical in geometry and differing in thickness only, as there is for ASTM **dogbone** specimens which differ in width only, it is necessary to use SL specimens with the same dimensions and geometry for all HDPE grades to obtain consistent, useful data from their TTs for a data base.

It is noted further that an SL specimen, with the same cross-sectional area in its GL as that of an ASTM specimen, did not produce load-strain curves that coincide with ASTM curves. It is concluded that caution must be assumed when attempting to interpret the SL results for comparison with dogbone results.

In the curves in Figure 7, the TTs are represented by solid-line curves. The circle-dash line curves were obtained from thermomechanical tests and are supplementary tensile data. The constant strain rate, uniform-strain cutoff locus is shown as a dash-line transversely intersecting the family of TT curves. The locus is obtained from the data for Figures 8a and 8b.

To the right of the locus, the data for strain rates and strain is no longer constant and uniform, respectively, and must be evaluated separately with care and caution.

The data from the curves in Figures 1, 7, 8, and 9 are included in Table 3 which is a compilation of specific engineering load-strain values (including those at the YP) picked from the curves of the material considered in this report. The data for each curve in Table 3 is from the lowest curve from a set of one to four curves. The variations in the load-strain data of Table 3 are due to the effects of geometry, cross section, crosshead speed, and temperature. Thus, Table 3 serves as a reference to, and for the comparison of, the effects of these parameters on the mechanical properties of these and other grades of HDPE.

In Figure 10, the GCC specimen was superior to the NCC in both stiffness (requiring a higher load to achieve a given strain) and YP for all the indicated temperatures and HSs.

The fast-cooled compression-molded material was superior to the container materials in strength and YP. The GCC material appeared to have been as strong as, and with the same YP as, the fast-cooled compression-molded material at 70°C with an HS of 0.05 in./min and only slightly weaker at 70°C with an HS of 5 in./min.

As far as is known, approximately 20% regrind was added to the virgin grade HXM 50100 HDPE by the container manufacturer who then made the NCC and GCC containers with the same molding process. Apparently, the tested container samples differ in color and mechanical properties due only to the addition of 4% green pigment.

Although all results are for HXM 50100 HDPE, the variations in the mechanical properties of the TT engineering load-strain curves in Figures 10a, 10b, and 10c are due to the processes used and the additives introduced. These variations are of sufficient magnitude to warrant treating their hosts as separate materials.

For all the materials of this report, almost without exception, the TT engineering curves for a head speed of 0.05 in./min, for both 20°C and 70°C, have a YP at an undetermined strain beyond 12% which was the extensometer test limit. However, for the head speed of 5 in./min, only the curve at 20°C has a YP (arrow) within the 12% strain.

The data in Figures 8a and 8b is from the simultaneous TT traces of load versus strain and load versus time. The straight-line portions of the log-log plots in Figures 8a and 8b are parallel to a reference line with slope equal to 1 indicating a linear relation between strain and time. Hence, in the straight-line portion of the plots to the cutoff strain, the average strain rate, e(t)/t, in the 1-inch GL is constant and uniform while the instantaneous strain rate is the same at each instant, so that  $\dot{e}(t) = e(t)/t = \text{constant}$ , where e is strain and t is time.

The data points show that, at the start of a TT for an HS of 5 in./min, ė(t) is higher than that on the straight line, but approaches the straight-line value rapidly, decreasing to the constant value within 100 milliseconds and within 1% strain. This initial region of nonconstant strain rate also occurs as an unstable zone in Figure 11. Why this occurs is not clear. After this initial inconsistency, ė(t) is uniform and constant to the cutoff. Beyond cutoff strain, ė(t) increases continuously to the YP. As the cutoff strain occurs for all of the specimen geometries tested, it is not considered a peculiarity of the SL specimen.

Table 3. TENSILE TEST ENGINEERING LOAD-STRAIN DATA AT THE INDICATED HDPE GRADES AND AT THE INDICATED CROSSHEAD SPEEDS AND TEMPERATURES

_Test	Tensile	Cross	Crosshead		Load (lbf) @	Indicated Stre	in e, (in./in.)	
Temp. (°C)	Specimen Shape	Section (sq. in.)	Speed, HS (in./min.)	e = 0.01	e = 0.02	e = 0.04	e = 0.06	YP Load (e)
) Sheet Gra	de (SG) - EHM-60	001						
21	SL	(1/8 x 1/4)	0.05	38.0	58.0	79.0	90.0	N/A
21	SL	(1/8 x 1/4)	5	N/A	N/A	N/A	N/A	N/A
21	SL	(1/8 x 1/4)	0.05	41.0	71.0	95.0	106.0	116.0 (0.14)
21	SL	(1/8 x 1/4)	5	72.0	110.0	146.0	153.0	163.0 (0.11)
21	SL	$(1/4 \times 1/4)$	0.05	82,5	121.5	158.0	175.0	185.0 (0.08)
21	SL	$(1/4 \times 1/4)$	5	103.0	173.0	235.0	255.0	263.0 (0.09)
21	Type IV	$(1/8 \times 1/4)$	0.05	42.5	62.0	81.0	90.0	99.0 (0.16)
21	Dogbone	(1/8 x 1/4)	5	62.5	97.0	126.0	136.0	141.0 (0,095
21	Type I	(1/8 x 1/2)	0.05	77.5	120.0	162.5	184.0	200.0 (0.12
21	Dogbone	(1/8 × 1/2)	5	129.0	200.0	257.5	277.5	285.0 (0.093
70	SL	(1/8 x 1/4)	0.05	7.5	13.0	22.5	28.0	N/A
70	SL	$(1/8 \times 1/4)$	5	N/A	N/A	N/A	N/A	N/A.
70	SL	(1/8 x 1/4)	0.05	10.0	18.0	29.0	35.0	N/A
70	SL	(1/8 x 1/4)	5	14.0	29.5	47.0	56.0	N/A
70	SL	(1/4 x 1/4)	0.05	21.5	34.0	49.0	58.0	N/A
70	SL	(1/4 x 1/4)	5	34.0	55.0	78.5	91.0	N/A
70	Type IV	(1/8 × 1/4)	0.05	6.0	14.0	25.0	32.0	N/A
70	Dogbone	$(1/8 \times 1/4)$	5	N/A	N/A	N/A	N/A	N/A
b) Blown-F	ilm Grade (BFG) -	HHM-TR140						
21	SL	(1/8 x 1/4)	0.05	23.0	41.0	60.0	69.0	N/A
21	SL	(1/8 x 1/4)	5	N/A	N/A	N/A	N/A	N/A
21	SL	(1/8 x 1/4)	0.05	32.0	39.0	63.0	80.0	95.0 (>0.2
21	SL	$(1/8 \times 1/4)$	5	45.0	71.0	98.0	112.0	116.0 (0.18
21	SL	$(1/4 \times 1/4)$	0.05	28.0	85.0	118.0	135.0	N/A
21	SL	$(1/4 \times 1/4)$	5	75.0	126.0	171.5	191.5	204.0 (0.1
21	Type i	(1/8 x 1/2)	0.05	54.0	82.0	114.0	132.0	N/A
21	Dogbone .	(1/8 x 1/2)	5	85.0	140.0	190.0	213.5	234.0 (0.11
70	SL	(1/8 x 1/4)	0.05	5.0	9.5	17.0	23.0	>156.0 (>0
70	SL	(1/8 x 1/4)	5	N/A	N/A	N/A	N/A	N/A

Table 3. (Cont'd) TENSILE TEST ENGINEERING LOAD-STRAIN DATA AT THE INDICATED HDPE GRADES AND AT THE INDICATED CROSSHEAD SPEEDS AND TEMPERATURES

	Test	Tensile	Cross	Crosshead		Load (lbf) @	Indicated S	Strain e, (in./i	n.)
Source	Temp. (°C)	Specimen Shape	Section (sq. in.)	Speed, HS (in./min)	e = 0.01	e = 0.02	e = 0.04	e = 0.06	YP Load (e)
Extra High Density P	olyethylene	- HXM 50100							
1. (NCC)	21	SL	(1/4 x 1/4)	0.05	60.0	91.0	125.0	141.0	235.0 (0.10)
	21	SL	(1/4 x 1/4)	5	80.0	137.5	192.5	216.0	N/A
2. (GCC)	21	SL	(1/4 x 1/4)	0.05	62.5	95.0	131.0	150.0	225.0 (0.10)
	21	SL	(1/4 x 1/4)	5	84.0	145.0	196.0	216.5	N/A
3. (NCC)	70	SL	(1/4 × 1/4)	0.05	22.0	34.0	50.0	58.0	N/A
	70	SL	(1/4 x 1/4)	5	35.0	55.0	79.0	91.0	N/A
4. (GCC)	70	SL	(1/4 x 1/4)	0.05	23.0	38.5	58.5	70.0	N/A
	70	SL	(1/4 x 1/4)	5	37.0	63.5	91.5	114.0	N/A
Cooling Plate Rate									

	Cooling									
Plate	Rate									
d) Compre	ssion Molded I	Polyethylen	e Plates of	Different Coolin	ng Rates - I	-IXM 50100				
ī	Slow	21	SL	$(1/4 \times 1/4)$	0.05	78.0	117.0	156.0	176.0	N/A
		21	SL	$(1/4 \times 1/4)$	5	108.0	170.0	222.0	243.0	260.0 (0.10
111	Medium	21	SL	(1/4 x 1/4)	0.05	75.0	113.0	154.0	175.0	N/A
		21	SL	$(1/4 \times 1/4)$	5	100.0	165.0	220.0	243.0	260.0 (0.10
11	Fast	21	SL	(1/4 x 1/4)	0.05	76.0	112.0	151.0	170.0	N/A
		21	SL	(1/4 × 1/4)	5	107.0	170.0	225.0	246.0	260.0 (0.10
ı	Slow	70	SL	(1/4 x 1/4)	0.05	28.0	44.Q	64.0	75.5	N/A
		70	SL	$(1/4 \times 1/4)$	5	42.5	72.0	105.0	123.0	N/A
111	Medium	70	SL	(1/4 x 1/4)	0.05	32.0	49.0	68.0	74.5	N/A
		70	SL	(1/4 x 1/4)	5	43.0	67.0	95.0	108.0	N/A
II	Fast	70	SL	(1/4 x 1/4)	0.05	23.5	43.5	61.5	71.5	N/A
		70	SL	$(1/4 \times 1/4)$	5	45.0	71.0	103.0	120.0	N/A

Note that tensile specimens, not necessarily of the same geometry, with equal cross-sectional areas in their GLs tend to have the same strain rate independent of the test temperature. As the cross-sectional area and/or HD(t) increases for each HS, the plots are displaced parallel toward longer times and lower strain rates.

Initially, the effective length, HD(t = 0), between grips is the actual length between grips which is 3, 4, and 2.5 inches for the SL, ASTM Type I, and ASTM Type IV tensile specimens, respectively, for the tests in this report.

Since the cross section may differ from point-to-point along HD(t), at any instant, all points in HD(t) do not strain uniformly, neither for ASTM-type dogbone or SL geometry. Furthermore, the material of the specimen is not homogeneous; being of two phases and also somewhat directionally oriented due to processing and perhaps containing voids, impurities, and even some molecular degradation due to processing. Thus, HS/HD(t) is an average strain rate in HD(t) which in turn is an average or effective length between the test grips. Likewise, e(t)/t and e(t) are average strain rate and strain in the GL.

Ignoring the first 1% strain, least squares analysis of the plots in Figure 11 indicates that HD(t) is independent of e(t)/t and e(t) in the GL to the cutoff strain (i.e. HD(t) is constant to cutoff). Thus, the average strain rate in HD(t) is equal to e(t)/t, the average strain rate in the GL, which is also constant and uniform in the GL (from Figure 8,  $\dot{e}(t) = e(t)/t = c$ ). Constant and uniform rate implies uniform strain so that the average strain in the GL is uniform. Indeed, all the specimens of the several geometries apparently strain uniformly in the GL as long as the GL strain rate,  $\dot{e}(t)$  and HD(t) are constant. The end of uniform strain is denoted by arrows at the curves' gentle knees in Figure 11.

The gentle knee indicates that the uniform strain cutoff conforms to the point of upward diversions from the straight line of a log e versus log t curve of Figure 8, indicating an increasing strain rate in HD(t), as well as in the GL beyond cutoff. The upward diversions are clearly evident on linear plots (not shown) of e versus t from which the strain cutoffs were obtained.

The knee, or cutoff strain, increases with test temperature; the larger increase occurring for the lower of the two HSs. The locus for cutoff strain is shown in Figure 7 superimposed on the TT curves for an HS of 0.05 in./min and several test temperatures.

Notice that beyond the cutoff, the GL strain becomes nonuniform, perhaps due to the nonhomogeneities in the form of flaws such as voids, and/or a crystalline-to-amorphous phase-change effect (see Figure 12, and associated Table 4). The GL strain continuously increases with load, becoming progressively more nonuniform while the strain rate to YP progressively increases due to stress concentration. Also, HD(t) decreases gradually beyond the knee in Figure 11.

# Table 4. PERCENT CRYSTALLINITY (% X) AND PERCENT CRYSTALLINITY CHANGE (% dX) OF HDPE (1/4 x 1/4) (SL) (ITT) (21°C) SPECIMENS AS A FUNCTION OF HEAD SPEED (BEFORE AND AFTER ITTs TO 12% ENGINEERING STRAIN)

High Density		Head Speed =	= 5.0 in./min.	Head Speed =	0.05 in./min.
Polyethylene (Grade)	% X Before Test	% X After Test	% dX	% X After Test	% dX
BFG	46.7	46.8*	0.1	46.8	0.1
NCC	52.1	50.0*	-4.0	49.5	-5.0
SG	54.6	53.5	-2.0	56.6*	3.66

<sup>\*</sup>Specimens X-rayed 13 months after TTs (remaining specimens X-rayed 6 months after TTs)

The authors were confronted with the surprising result that in the flat tensile specimens of the present material the GL strain was uniform only to a cutoff strain which was well below the YP. Likewise, the GL strain rate was constant only to the cutoff, beyond which it increased continuously, reaching a maximum value at the YP. The effect was surprising because constant strain rate and uniform strain in the GL is widely believed, and reported in the literature, to hold to the maximum load where incipient necking suddenly occurs. It appeared that nonuniform strain occurred much sooner at cutoff; then localized, gradually increasing and coalescing with other localized strains, culminating in a shear band at the maximum load. Likewise, the strain rate continuously increased beyond cutoff due to localization of strain reaching a maximum in the shear band.

TT curves, like those in Figure 12, are obtained when (1/4 x 1/4)(SL) specimens are tested at 21°C and stressed to 12% strain which is beyond YP strain for an HS of 5 in./min and before it for an HS of 0.05 in./min. As these curves are from specimens with the same shape, thickness and GL cross-sectional area, they give a clear indication that the container material is of different morphology than both SG and BFG HDPE. This difference was verified by GPC (see Table 1) and X-ray diffraction analysis with which differences in molecular distribution (not shown) and differences in percent crystallinity, respectively, for the three grades are observed. Creep curves at 70°C and 250 psi for the three grades also show a clear difference for a one-hour DL test performed with (1/4 x 1/4)(SL) specimens (curves not shown).

The X-ray diffraction analysis was performed in transmission<sup>26</sup> in the GL of the (1/4 x 1/4)(SL) specimens. The X-ray analysis which has a 2% experimental precision and a 5% systematic error, indicated no residual stresses and no orientation in the crystalline phases in any of the HDPE grades, neither before nor after a TT. The X-ray diffraction procedure used by Reference 26 is given in Reference 27.

However, as indicated previously in this report, the injection-molded specimens consisting of bar thicknesses (from which SL specimens were routed), contained overall shrinkage as well as concave dishing in the specimen faces. This and the X-ray results seem to indicate that any residual from these effects is in the amorphous phase of the HDPE as is any molecular orientation due to the injection molding process.

After tensile testing the three grades of HDPE to 12% strain at  $21^{\circ}$ C and crosshead speeds of 5 and 0.05 in./min, they were examined by X-ray diffraction for percent-change in crystallinity. Analysis of the X-ray data showed no affect on the crystallinity for all three grades after the testing when compared to a set of untested specimens, recalling the work of Moore and Matsuoka<sup>6</sup> (see the Discussion Section). The X-ray tests were performed on three of the tested specimens six months after the tensile tests and on the remaining three (identified by asterisks in Table 4 where all six specimens are tabulated) 13 months after their tensile tests. The results appear to indicate no long-term room temperature crystal recovery or annealing in the specimens which were stored in polyethylene bags at 50% relative humidity and  $21^{\circ}$ C  $\pm$  4°C in a covered, five gallon, white PE bucket prior to X-ray analysis.

Figure 6 shows the results of a 42-day total creep strain versus real-time tensile creep test performed on a HDPE (1/4 x 1/4)(SL) specimen routed from material cut from the NCC with the creep apparatus described earlier. The total creep strain consists of the loading strain plus the transient creep strain. The curve shows that the transient strain (initial response immediately after full DL is applied) under DL apparently ceases after roughly five days with a 1% total creep strain at the start of the creep-strain plateau at 250 psi and 70°C. Thus, after five days, the creep ceased with a total strain of 1% and remained that way until the 42nd day.

Upon unloading at 70°C on the 42nd day of creep, most of the strain recovered in six days. A large part of the unrecovered strain, if not all, is presumed to be due to the experimental strain error. Any further recovery is expected to be small.

Thus, shortly after loading, the material appears to cease to creep (a plateau) but does not recover completely upon unloading. The data of Gohn and Cummings<sup>28</sup> indicates that their PE creep tests took years to reach this plateau state, if at all (see the Discussion Section).

Further experimentation, taking into account the systematic errors in the creep apparatus, is needed to resolve the question of complete recovery (see further details in the Discussion Section, particularly about plateau recovery results for polycarbonate).

Figures 4 and 5 contain low-load tensile creep curves, at  $20^{\circ}$ C and  $70^{\circ}$ C, unloaded after 10 to 20 hours of creep. The tests were performed to show the existence of full recovery and, hence, the existence of LVE conditions around and below the critical stress. Although plateaus occurred, none was found from which full recovery occurred so that LVE conditions could not be conclusively established. The best that could be obtained was **near complete** recovery which, once again, is believed to point to the systematic error inherent in the creep apparatus. Thus, although some curves have the appearance of full recovery in the figures, all of the recovery strains in Figures 4 and 5 have at least a  $\pm 0.0005$  unrecovered strain (also shown in Figure 6). The dashed area (see Figure 4a) indicates a case of over recovery due to the systematic errors.

It was expected that creep from a 145 psi (1 MPa) stress at 20°C will plateau sooner and recover more fully than that from the same stress at 70°C. However, the large capricious strain error found accompanying the loading and unloading (described in the test procedures) tended to render moot the question of the existence of complete recovery at this time. One may question the plateau as an artifact due to a stuck apparatus. It is believed this is not

the case as there was an unobstructed, transient creep-strain effect gradually leading to the plateau, most likely due to the viscoelastic material and not apparatus friction.

Intrinsic Creep Loads and Specimen Heating at Constant Temperature Rate

Once again, the region being studied involves small DLs (stresses) and strains. Although this is the region of creep testing where the creep results with external DLs are affected by the bending moment and other limitations in the creep apparatus, none of these affect the measurement of the thermal strain which involves the coefficient of expansion and the ever-present intrinsic loads for the reasons mentioned in the Creep Test Equipment Section.

Intrinsic loads, imparted in this case by the weights of the lower half of the extensometer and a lower grip, produce an unintended and undesirable premature strain on the mounted test specimen, inadvertently shifting the room temperature equilibrium of the test specimen to a new one slightly removed from that of the load-free specimen, by producing a small, unmeasurable (unknown) strain. This strain was set to zero along with the thermal strain effect of  $\alpha$ , the coefficient of linear thermal expansion, at room temperature on a strain versus time chart when the extensometer zero strain is set prior to a test. The intrinsic loads were considered as parts of the total load, although the strain of the intrinsic loads was zeroed out, and to that extent were a source of total creep-strain error particularly for small total loads (the total load contains the intrinsic and external DLs), as shown in Figure 13.

As parts of the small total load, the intrinsic loads w(i) contribute to the total creep strain. Considered by themselves, their presence is visible through the temperature dependent part of their strain e[w(i),T] above (and below)  $20^{\circ}$ C, where their unknown  $20^{\circ}$ C strain was set to zero as an extension (or a contraction) of the material, additional to the thermal-strain effect of  $\alpha$ . Upon return to  $20^{\circ}$ C, the expansion or contraction of a material becomes zero since the strain due to the intrinsic loads and temperature on the material is initially set to zero here. Of course, this is possible only if the experimental error of the apparatus is negligibly small and the material is LVE with no morphological changes in it.

Consider Figure 13, which is a schematic diagram of total strain versus time and temperature at constant heating rate with total **external** creep strain due to external DL, w(ex), superimposed on thermal strains due to w(i) and  $\alpha$ . As indicated earlier, there are limitations in performing this type of experiment for HDPE with a small w(ex) (with small stresses  $\leq 350$  psi) without avoiding error due to the limitations of the creep apparatus. However, it is possible to perform it when the external load is zero [w(ex) = 0] so that only intrinsic loads exist. Note that the form of the curve in Figure 10 does not occur in the usual creep test since the thermal components are zeroed out with the extensometer. An example is at  $70^{\circ}$ C before the external DL is applied. It is at this point that the usual creep test begins (as shown in Figures 4, and 6).

As all creep tests in this report contain the intrinsic loads, it is useful to know how they affect the test. To do this, only this load was used for examination and testing.

Figure 14 illustrates the thermal strain curve,  $\Delta(T, t)$ , due to the intrinsic load, w(i), and  $\alpha$  when the HDPE specimen is heated and, after a while, cooled. The test was performed with an (NCC)(1/4 x 1/4)(SL) tensile specimen with a w(i) of 2.7 lbf (12 newton, N) so that the stress was 43 psi. The specimen was heated and cooled at the rate of 2°C per minute.

Because the experimental heating and cooling curves are straight lines [although a slight inflection point at approximately 50°C (not obvious in Figure 14) may be indicative of a transition point<sup>29</sup>], we assume a linear relation for  $\Delta(T, t)$  and its components so that  $\Delta(T, t) = \Delta e + e[\alpha(T)]$ .

To facilitate a separation of the strain into its components, the value of  $\alpha$  is assigned, which is equal to  $110 \times 10^{-5}$  in./in. $^{\circ}C^{30}$  and it is proposed that it be constant over the heating and cooling range of  $20^{\circ}C$  to  $70^{\circ}C$ . Thus, there are two components of strain:  $\Delta e$  and  $e[\alpha(t)]$ , where  $\Delta e$  is the shaded portion of the curve in Figures 13 and 14. It can be seen that  $e[\alpha(T)]$  and  $\Delta e$ , which were zeroed out at  $20^{\circ}C$  when the extensometer was set to zero strain, are temperature dependent so that  $e[\alpha(T)] = 2 \Delta e$  ( $\Delta e = 0.002$  in./in. on the  $70^{\circ}C$  plateau) above (and below)  $20^{\circ}C$ . Also, both are fully reversible approaching zero strain as the temperature approaches  $20^{\circ}C$  (from above or below). If w(i) was 0 to start, then only  $e[\alpha(t)]$  would be involved (unshaded part) and the test would be a thermomechanical or a thermodilatometric test. Several things are observed with constant  $\alpha$ :

- 1. For a specimen with an initial cross section,  $A_0$ ,  $\Delta e$ , and  $e[\alpha(T)]$  are independent of T for a T of 1° or 2°C/min.
- 2. Further, if we assume that  $\alpha$  is independent of  $A_0$ , then we assume that only  $\Delta e$  depends on  $A_0$ .
- 3.  $\Delta e$  is fully reversible as is  $e[\alpha(T)]$ . Note that  $\Delta e$  would be zero if there were no w(i); then, prior to a test, the extensometer would zero out only  $e[\alpha(T)]$ . Note that in a TT at, for example,  $70^{\circ}$ C, the load cell and extensometer zero out any preload strain and  $e[\alpha(T)]$  prior to a test, as was described for the creep extensometer above.

In Figure 14, it is not apparent which component is affected by the seven-hour crystal growth seen as the strain decrease,  $\Delta x = -0.021\%$ , which remains constant through a 30-minute cooling to  $20^{\circ}$ C and for 15 hours after.

Another question is whether the crystal growth would continue beyond seven hours and, if so, how much and for how long?

There is the further possibility that  $\Delta x$  is due to annealing residual strain in the amorphous phase of the HDPE (NCC) material so as to cause the small, negative-strain adjustment and not involve crystal growth.

# **DISCUSSION**

A comprehensive study was made of the mechanical behavior (tensile and creep) of three grades of HDPE over a range of load and temperature. The results are included to serve as a reference on the mechanical properties of the three grades of HDPE treated herein and as a reference for future work on them and other HDPE grades. Figures 1, 7, 8, 9, 10, 11, and 12, along with all of the tables, are used to establish a data base on the three HDPE grades.

In Figures 8, 11, and 12, it was found that the  $20^{\circ}$ C strain is nonuniform beyond 3% in a tensile test with flat specimen (as well as in other mechanical tests, such as the creep test) and that the strain rate is constant to only 3% beyond which it increases monotonically to the yield point. To help explain these findings, refer to the room temperature findings of

Moore and Matsuoka<sup>6</sup> with Marlex 6000-60 (Phillips 66 Company) which they described as a medium molecular weight linear PE with an Mw of 40,000, density of 0.96 g/cc, and 80% crystallinity. They observed a scattered maser-light pattern, from a PE film specimen deforming in the tensile creep mode, to broaden with strain. The broadening, according to them, is due to cumulative deformation in spherulites where "The first indication of deformed spherulites is detected at about 3% strain though some deformation probably occurs before this."6 Thus, the deformation in the spherulites increases with strain. Its collective effect became evident at 3% strain as a broadening of the light pattern from a deforming creep specimen, also appears to be related to the start of nonuniform strain and the end of constant strain rate in the TTs, as seen in Figures 8 and 12. Upon unloading from a 1740 psi (12 MPa) constant stress with a 6% tensile creep strain, after about five hours, Moore and Matsuoka found the broadening in the pattern gradually disappeared and the pattern returned to its original state, but that full strain recovery did not occur. This also occurred in a similar experiment by Erhardt et al.<sup>31</sup> Since the X-ray findings on tested tensile specimens (see Figure 12 and Table 4) indicate no loss of crystallinity or change in its X-ray pattern from loads producing strains of 12%, it is suggested that the light-pattern change is produced by spherulite strain which is part of the elastic and linear viscoelastic strain components with the viscous strain component apparently residing in the amorphous phase not playing any major part in pattern change. The full recovery of the pattern is due to full recovery, with time, of the spherulite strain of the elastic and linear viscoelastic strain components (the remainder is due to the amorphous phase) of the creep loading. As the stress is much greater than the Nielsen critical stress, a large viscous strain component occurred as part of the total, 6% creep strain which upon unloading remained unrecovered in the amorphous phase.

Turning now to the low-load creep experiments, a literature search was performed to get experimental support for the creep strain and recovery results. The desired support would be in the form of creep at low loads for several temperatures including room temperature and those above and below it and, at least, implies a critical stress. Only partial support was found.

Recall that the trend in the shapes of the individual creep curves in any family of such curves is towards a flattening of the curves (plateauing) with decreasing stress at constant test temperature or increasing temperature at constant stress. It is expected that the test curves should approach a plateau and full recovery as the irrecoverable, viscous creep strain approaches zero at the critical stress. For all stresses below the critical stress, LVE results are expected; i.e., a creep strain which reaches a constant value (a plateau with no strain rate or viscous creep strain) and, upon unloading, full creep-strain recovery at any temperature (barring structural changes in the material).

In work on PE pertinent to our plateau concept, "Lethersich<sup>32</sup> shows that with shear stresses up to . . . 150 psi the initial elastic strain and the primary creep (that is, the total deflection less the steady-state creep, if it exists) are linear with stress" (quoted from the chapter by Hopkins and Davis<sup>33</sup>). (Bold lettering to the above referenced material was added by the authors of the present report).

Some of the earliest work on creep of PE was by Gohn and et al.  $^{28,34,35}$  who tested several grades of PE (one natural, all others with added carbon black of less than or equal to 2%) for periods of up to 11 years with low loads down to 0.345 MPa (50 psi) at  $30^{\circ}$ C  $\pm 1^{\circ}$ C. Their conclusion was that all of the specimens experienced creep that diminished and ceased with time. The time interval for creep cessation decreased as the load (stress)

decreased and the creep became less than the precision of measurement up to 2.76 MPa (400 psi). Nielsen<sup>7</sup> placed this data in the subcritical stress region. Indeed, for stresses less than or equal to 0.69 MPa (100 psi), in the Gohn data, <sup>28</sup> one can argue to linear viscoelastic conditions for some of their material (through log stress versus log strain isochrones of 1000 hours) although this material did not recover completely even after 600 hours.

Findley<sup>36,37</sup> performed a creep test on a single specimen of PE under interrupted loading (unload/reload in the 16th year) for 26 years at 225 psi and 25°C. The shape of the curve suggests that the stress for his material was above critical stress as there was no cessation of creep; even so, the magnitude of the viscous creep strain is relatively small suggesting that the stress was not too far above the critical stress.

Crissman<sup>38</sup> performed short-time creep tests on HDPE at 20°C, 38°C, and 50°C and mostly at 4 MPa (580 psi). From his results, he concluded that there was no linearity, even at his lowest load of 1 MPa with strain of 0.1%.

As to the question of what constitutes linearity in a viscoelastic material, Ward and Onat<sup>39</sup> presented a criterion for LVE by plotting creep compliance against stress, the material being linear so long as compliance was independent of stress. While others (Turner<sup>40</sup> and Crissman<sup>38</sup>) used the viscoelastic reference line, dlog(stress)/dlog(strain) equals 1, plotting log stress versus log strain isochrones at selected constant times. The isochrones indicate LVE conditions if they are parallel to the reference line.

Nielsen states that "the critical stress is often about the same value as the stress at which the stress-strain curve becomes nonlinear."8

However, for HDPE creep tests done well below the critical stress; i.e., at a stress less than or equal to 1 MPa where one might expect a plateau and complete recovery, large scatter was found, as well as inconsistencies and poor reproducibility leading to the conclusion that the lower limit was reached with the apparatus for producing and reproducing scientifically reliable creep-recovery data (see Figures 4 and 5) and we would not be able to pursue the plateau concept with this apparatus.

To overcome this difficulty, one test procedure is to use simple, low weight but strong specimen grips, and to avoid intrinsic preload and bending moments on the specimen by replacing the present creep extensometer with two resistance foil strain gages directly attached with proper adhesive to opposite faces of the GL of the test specimen. This technique was not readily available for HDPE since the available adhesives (e.g., epoxy) do not stick to it. However, there are techniques to make epoxy work, one being a 10-minute HDPE specimen exposure to a low temperature oxygen plasma. As this is beyond the scope of this study, polycarbonate (PC) was used to demonstrate the existence of a creep-strain plateau and the subsequent full recovery from it upon unloading.

A (PC)(1/8 x 1/2)(bar)(21°C) with resistance strain gages glued with epoxy directly opposite each other onto opposite flat sides of the 5.5-inch bar is tested at low loads using the techniques described above for LVE properties. LVE properties are obtained in the PC bar manifested by creep-strain plateauing with subsequent full strain recovery upon unloading at any time on the plateau. The results were successful for three stress levels (85, 350, and 700 psi) each of which resulted in creep-strain plateaus from which full recovery was obtained. The specimen loading/unloading is done with a manually controlled, smoothly

operating scissor-type platform suited for handling small loads. The same specimen was used for the three aforementioned stress levels. Each recovery was complete in about 10 minutes. Apparently, the specimen had no memory of each prior test. This effect is a corollary of our definition of LVE conditions in a plastic.

This test data compares favorably to the LVE aspect of PC unpublished creep data<sup>42</sup> at 21°C which indicated plateaus and full recovery between 10 MPa (1450 psi) and 22.5 MPa (3270 psi) which, in turn, compares favorably to creep data of Yannas and Lunn.<sup>43</sup> The data of Reference 42 was obtained from tests performed with the creep apparatus described in this report. As the testing was done well outside the error zone, the error in strain due to the enumerated sources of error is negligible for these tests.

In light of the creep plateau recovery results on medium viscosity, amorphous PC (Lexan), HDPE probably has a LVE range manifested by plateauing of the creep strain, fully recovering upon unloading, for stresses below a critical stress. Corroberation will probably be obtained with a test like the one described above for PC once the strain gage adhesive problem is overcome. Then, also, an attempt would be made to establish the critical stress and its relation to temperature.

#### CONCLUSIONS

Each grade of HDPE, along with its processing, has its own mechanical properties characteristics. To that extent, each should be treated as a separate material with its own data base.

The AMMRC streamline specimens produced a different response to mechanical tensile loading from that of ASTM-type specimens so that care must be exercised when comparing their mechanical performance to that from ASTM-type specimens and, presumably, from specimens with other geometries.

In a TT, the strain tends to be uniform with a constant strain rate in HD(t) (the effective length between grips) and both the uniform strain and constant strain rate in this HD(t) cease at approximately 3% at 21°C and at approximately 7% at 70°C. Beyond this cutoff point, nonuniform strain develops and the strain rate increases monotonically approaching the HS, culminating in a shear band in the GL at maximum load (yield point).

X-ray analysis of HDPE TT data shows that the nonuniform strain and monotonically increasing strain rate beyond 3% to 12% (yield point) strain is not related to any change in percent crystallinity at room temperature.

The creep-strain plateau full-recovery concept, along with the measurable, finite loading time and the increasing strain during DL application, could be of importance to fundamental concepts in linear viscoelasticity. However, the creep apparatus is found to have an experimentally unmanageable strain zone for obtaining scientifically acceptable HDPE creep-recovery data due to the capricious bending moments occurring when a small external DL is applied or removed. This affects the conclusive establishment of full recovery from a plateau for all stress levels below a critical stress level and its relationship to linear viscoelasticity. This difficulty is expected to be overcome once the problem of gluing strain gages with adhesive wetable to HDPE onto specimen faces is solved. The proposed experimental arrangement toward this end requires a sensitive means of recording creep strain and recovery while eliminating the intrinsic load and specimen bending moments caused by application of the external loads.

In the meantime, PC, with attached strain gages and DL stresses, was used as a surrogate for establishing full recovery from a plateau. Preliminary results at low loads along with unpublished PC creep-recovery data indicate plateaus for several stresses and subsequent full recovery at 20°C showing LVE conditions. A critical stress level is expected at some higher loading level along with cessation of LVE properties of the PC specimen. Unpublished PC data places this level roughly at 3300 psi at 20°C with a 1% strain.

Until materials characterization is available, in the sense of tailoring the material to fit the job, one must continue to fit the job to the available shelf material relying on its data base for its design properties. To present the best possible data base to this end, the best possible documentation of all facets of the material from raw material inception to its processing and testing, along with incidental treatments, is absolutely essential.

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Our thanks to the following personnel at the U.S. Army Materials Technology Laboratory: J. Connor for the injection-molded specimens, P. Dehmer for helpful discussions on polyethylene in general, R. E. Pasternak and A. L. Fuller for technical assistance with the TTs in many instances, C. Cavallaro for mounting strain gages on the polycarbonate sample and for technical assistance on the electronics for the creep strain-strain recovery experiments with polycarbonate, D. Dunn for the HDPE molecular weight data by gel permeation chromatography, D. P. Macaione for the thermogravimetric data on HDPE and PC, H. T. Laye for the line drawings with many improvements, L. Caruso for typing the first draft of this report from dictation, and S. Sarian for several rewrite passes on the computer.

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## REFERENCES

- 1. WIGOTSKY, V. Plastics Engineering. 27 April 1987.
- 2. WIGOTSKY, V. Plastics Engineering. 27 April 1989.
- 3. TRANTINA, G. G. Polym. Eng. Sci., v. 26, 1986, p. 776.
- 4. TOBIN, W. Plastics Design Forum, v. 59, March/April 1988.
- 5. OPLINGER, D. W., PARKER, B. S., GHANDI, K. R., LAMOTHE, R., and FOLEY, G. ASTM STP 864, 1985.
- 6. MOORE, R. S., and MATSUOKA, S. J. of Polym. Sci., Part C, No. 5.
- NEILSEN, L. E. in Mechanical Properties of Polymers. Rheinhold Pub. Corp., New York, 1962.
  NIELSEN, L. E. in Mechanical Properties of Polymers and Composites, Marcel Dekker, Inc., New York, v. 1, 1974.
- 9. DIETER, G. L. in Mechanical Metallurgy, 2nd edit., Section 8.2, McGraw-Hill, New York, 1976.
- 10. ALDEN, T. H. Metall. Trans. A., v. 16, 1985, p. 375.
- 11. ALDEN, T. H. Mat. Sci., Eng., A., v. 103, 1983, 213.
- 12. Forest Green Color Concentrate, PMS Consolidated Co., NJ.
- 13. Wabash 100, Wabash Metal Products, Inc., Wabash, IN 46992.
- 14. ASTM Standard, 802, Test Method D 1928-80, 1987.
- 15. ASTM Standard, 801, Test Method D 1248-84, 1987.
- 16. HAGNAUER, G. Personal Communication. OML-MTL, Watertown, MA 02172.
- 17. Van Dorn 200-RS-20F, Van Dorn Plastic Machinery Co., Cleveland, OH 44104.
- 18. ASTM Standard, 801, Test Method D 638-86, 1987.
- 19. Applied Test Systems, Inc., Butler, PA 16001.
- 20. Associated Environmental Systems, Lawrence, MA 01842.
- 21. Pickering and Co., Inc., Plainview, NY 11803.
- 22. Metals Handbook, Mechanical Testing, 9th Edition, ASM, OH, v. 8, 1985.
- 23. Allen Datagraph, Salem, NH 03079.
- 24. WARNAS, A. A. To be published.
- 25. Instron Corp., Canton, MA 02021.
- 26. DESPER, C. R. MCD-USA-MTL, Watertown, MA 02172.
- 27. DESPER, C. R. U.S. Army Materials Technology Laboratory, AMMRC TR 84-28.
- 28. GOHN, G. R., and CUMMINGS, J. D. ASTM Bulletin, July, 1960.
- 29. HU, S.-R., KYU, T., and STEIN, R. S. J. Polym. Sci., Part B, Polym. Phys., v. 25, 1987, 71.
- 30. Plastics Encyclopedia, 1985.
- 31. ERHARDT, P., SASAGURI, K., STEIN, R. S. J. of Poly. Sci., Part C, No. 5.
- 32. LETHERSICH, W. Proc. Intern. Cong. Pure and Applied Chem., 11th Congress, London, 1947, v. 5, 1953, 591.
- 33. HOPKINS, I. L., and BAKER, W. O. Rheology, F. R. Eirich, ed., Academic Press, New York, v. 3, chpt. 10, 1960.
- 34. GOHN, G. R., CUMMINGS, J. D., and ELLIS, W. C. Proc., ASTM, 49, 1949, 1139.
- 35. GOHN, G. R., ARNOLD, S. M., and BOUTON, G. M. Proc., ASTM, 46, 1946, 990.
- 36. FINDLEY, W. N., and TRACY, J. F. Polym. Eng. Sci., v. 14, 1974, 577.
- 37. FINDLEY, W. N., Polym. Eng. Sci., v. 27, 1987, 582.
- 38. CRISSMAN, J. M. Polym. Eng. Sci., v. 26, 1986, 1050.
- 39. WARD, J. M., and ONAT, E. T. J. Mech. Phys. Solids, v. 11, 1963, 217.
- 40. TURNER, S. Pohm. Eng. Sci., v. 6, 1966, 306.
- 41. PETRIE, E. M. Adhesive Age, 15 May 1989.
- 42. WARNAS, A. A. Unpublished PC-Data, 1985.
- 43. YANNAS, I. V., and LUNN, A. C. Macromol. Sci.-Phys., B4, v. 3, 1970, 603

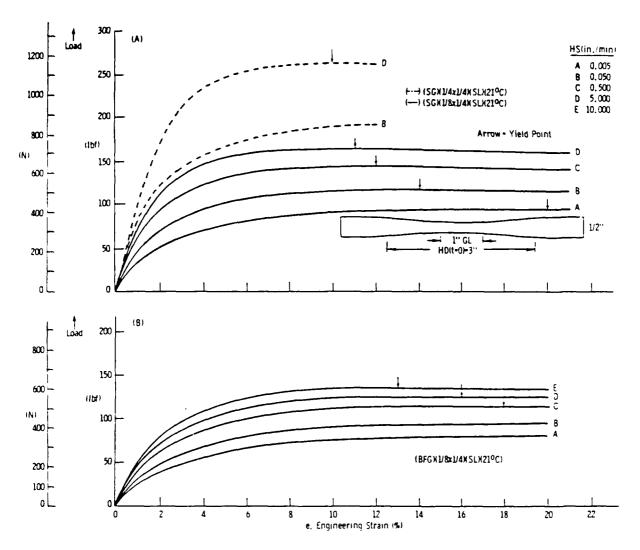


Figure 1a. TTs on HDPE SL-specimens of SG and BFG at 21°C and the indicated HSs produced the families of engineering load-strain curves. Figure 1b, doubling the cross-sectional area in the GL does not result in a doubling of the load at a given strain for a given temperature and HS.

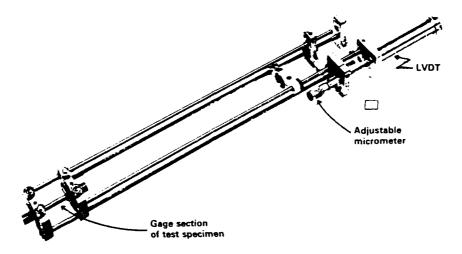


Figure 2. Typical rod-and-tube-type extensometer for elevated-temperature creep testing.



Figure 3. Linear variable differential transformer.

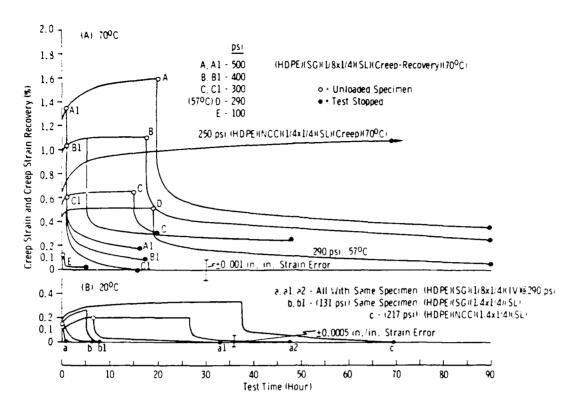


Figure 4. Total creep strain-recovery curves at the constant DL (stresses) and temperatures indicated with error bars showing why complete recovery could not be achieved.

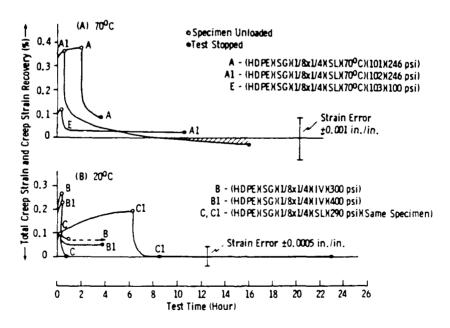


Figure 5. Load/unload total creep strain curves showing strain recovery at 70°C and 20°C for the HDPE grades and specimens indicated, and at the indicated stress levels. The problem of indicated stress levels. The problem of determining if full recovery occurred is illustrated by the size of the strain errors which prevents determining LVE properties.

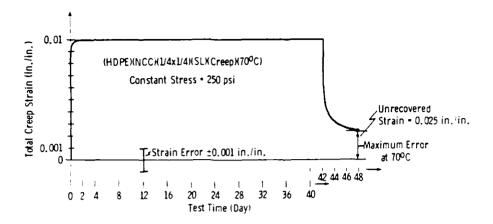


Figure 6. Total creep strain and recovery strain versus test time after 42 days creep and six days recovery at constant stress of 250 psi at 70°C.

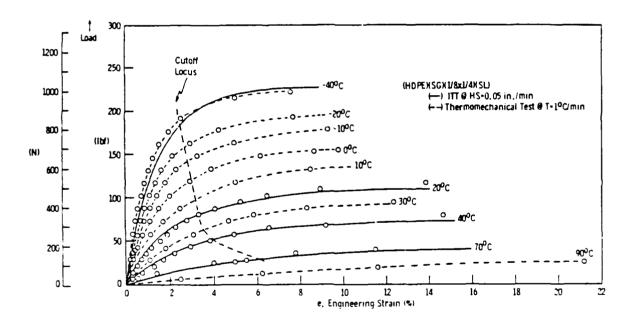


Figure 7. TTs on (HDPE)(SG)(1/8 x 1/4)(SL) specimens at 0.05 in./min and the indicated test temperatures produced the families of engineering load-strain solid-line curves. The dash-line curves and/or circle curves are constructed from thermomechanical test data. They are included as supplementary curves. The cutoff locus, which cuts the family of load-strain curves transversely, indicates two zones for strain and strain rate: to the left of the locus, the strains are uniform and the strain rate on any curve is constant; to the right, the strain is nonuniform and the strain rate increases monotonically to the YP along any load-strain curve.

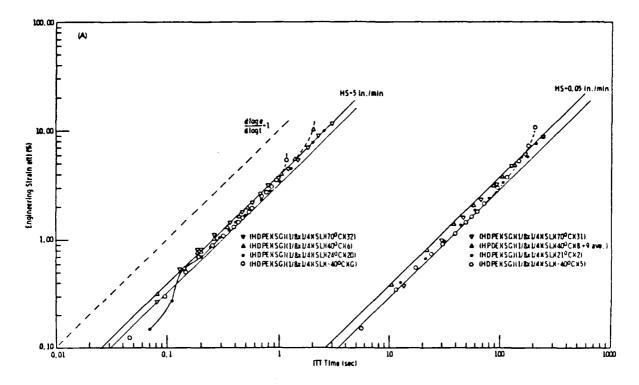


Figure 8a. Log-log plot of TT engineering strain, e(t) versus time, t for (HDPE)(SG) (1/8 x 1/4)(SL) specimens tested at the indicated HSs and temperatures.

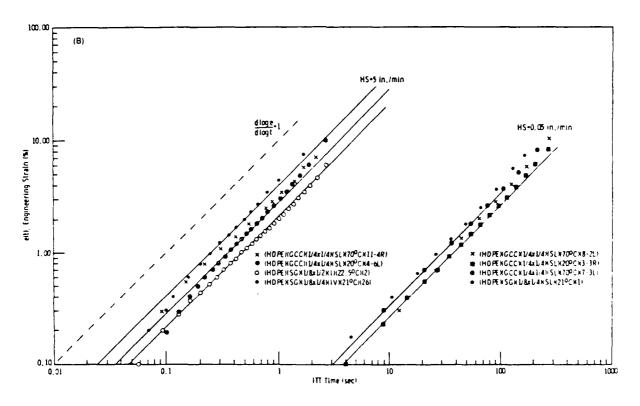


Figure 8b. Log-log plot of TT engineering strain, e(t) versus time, t for several grades of HDPE and specimen types tested at the indicated HSs and temperatures.

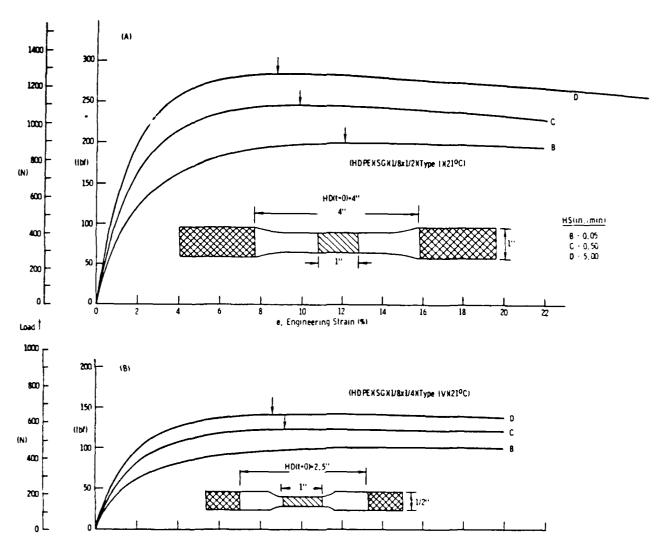


Figure 9. TTs on HDPE ASTM Types IV and I dogbone specimens of SG at 21°C and the indicated HSs produced the families of engineering load-strain curves in Figures 9a and 9b. A doubling of the load at any given strain, for a given temperature and HS, in Figure 9b places the load-strain point on the corresponding curve in Figure 9a, at the same temperature and HS, up to the YP.

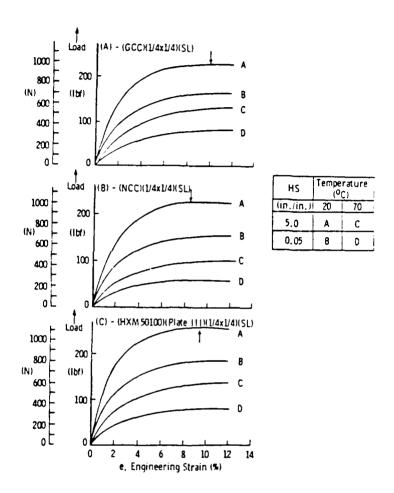


Figure 10. TTs on HDPE HXM 50100 processed in three different ways at the indicated HSs and temperatures produced the families of engineering load-strain curves. The materials are processed as: Figure 10a, GCC; Figure 10b, NCC from blow-molded containers, Figure 10c, from compression-molded Plate III.

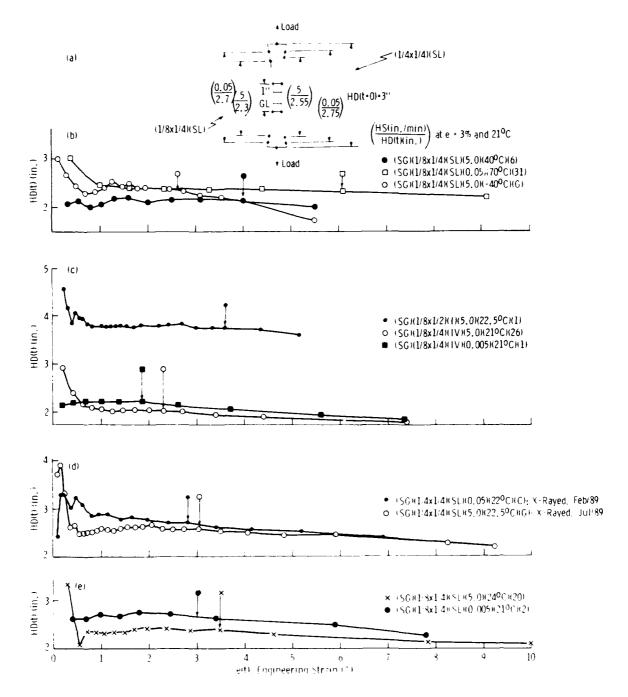


Figure 11. The effective specimen length HD(t) versus e(t) at time, t for HDPE SG at the indicated temperatures and HSs and for several specimen geometries. Figure 6a shows HD(t) at e(t) = 3% at  $21^{\circ}$ C for the HSs of 0.05 and 5 in./min and for two cross sections. The arrow at any curve knee shows approximately the end of uniform strain and constant strain rate.

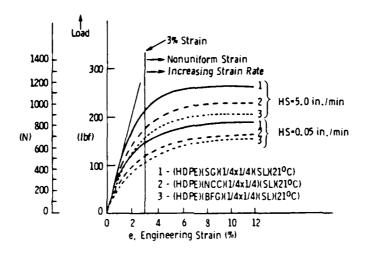


Figure 12. TT engineering load-strain curves of three grades of HDPE produced at 21°C and indicated HSs. The specimens producing these curves were X-rayed. It was found, in all cases, that these specimens had only minor crystallinity changes due to straining. The results are shown in Table 4.

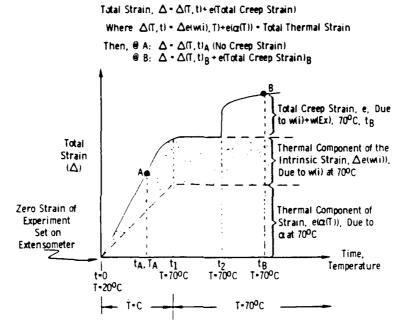
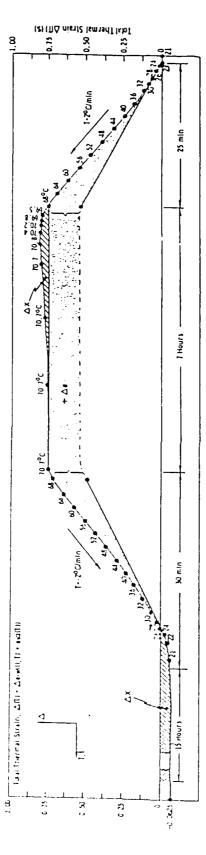


Figure 13. A schematic diagram with total creep strain due to w(ex) superimposed on the thermal components of strain due to a, the coefficient of linear expansion, and the intrinsic loads, w(i) at  $70^{\circ}$ C.



and cooled at  $2^{\circ}C/\min$ . The thermal strain is separated into strain components  $\Lambda$ e due to a constant, intrinsic load  $M(t) = 2^{\circ}C/M$  by and e( $\alpha = 90 \times 10^{\circ}$  in /(in.  $^{\circ}C)$ . Both components are temperature dependent and proportional to temperature; e( $\alpha = 10^{\circ}C$ ) and material experiences annealing of residual stress and/or crystal growth manifested as a decrease in total thermal strain by  $\Lambda X = 0.021\%$  at 70°C which is fully preserved upon cooling to 20°C in 30 minutes and for 15 hours after. Figure 14. Thermal strain due to a small, intrinsic load w(i) and a the coefficient of linear expansion, on (HDPE) (NCC) (1/4 x 1/4) (SL), heated

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CREEP TESTS - Albert A. Warnas and Marc S. Pepi PROPERTIES OF HIGH DENSITY POLYETHYLENE **OBTAINED FROM TENSILE AND DEAD-LOAD** MECHANICAL AND LINEAR VISCOELASTIC U.S. Army Materials Technology Laboratory Watertown, Massachusetts 02172-0001

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Mechanical properties **Key Words** Polyethylene

Linear viscoelasticity (LVE)

Three grades of high density polyethylene (HDPE) are tested for tensile, creep and creep recovery, and creep properties determination. As a result of tensile tests, the materials were found to strain uniformly under constant attain rate to about 36 at 20°C which is well below the 12% yield point (FV). With creep tests, an attempt was made to explain and predict the effects of low loads and femperature on linear viscoelasticity (LE) in these materials. When a creep curve plateaus (zero creep plateau is 10°Cs. This combination of creep plateau and full recovery as another manifestation and definition of hiear viscoelasticity is presented. The effort presented was only partially successful for HDPE as the creep apparatus has an error zone producing uncertain data presented supparatus, it was found that amonothous polycustomate (PC) produces plateaus except on the same creep apparatus, it was found that amonothous polycustomate (PC) produces plateaus outside and above this error zone, from which full recovery occurs at strains up to 1% and stresses up to approximately 3300 psi (22.75 MPa). An engineering load strain data base is established for the three grades of HDPE tested using the U.S. Army Materials and Mechanics Research Center (AMMRC) streamline tensile specimens. A comparison is made to the load-strain results from two types of ASTM where seemed streams. It is noted the AMMRC streamline tensile specimens have a separate response to mechanical tensile loading from tential of ASTM-type specimens so that care must be exercised when comparing their mechanical performance. mance to that of ASTM-type specimens

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